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IS 8707 (1978): Mancozeb, Technical [FAD 1: Pesticides and Pesticides Residue Analysis]

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भारतीय मानक

मेनकोजेब, तकनीकी — विशिष्टि

(पहला पुनरीक्षण)

Indian Standard

MANCOZEB, TECHNICAL — SPECIFICATION

(First Revision)

ICS 65.100.30

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

February 2013 **Price Group 5**

Pesticides and Pesticides Residues Analysis Sectional Committee, FAD 1

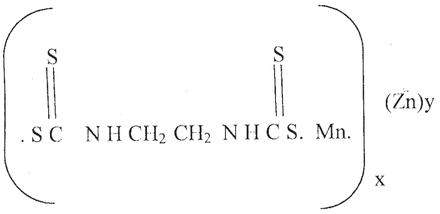
FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Pesticides and Pesticides Residues Analysis Sectional Committee had been approved by the Food and Agriculture Division Council.

Mancozeb is a protective fungicide effective against a wide range of foliage diseases of plants.

Mancozeb is the accepted common name by the International Organization for Standardization (ISO) for a co-ordination product of manganese ethylene bisdithiocarbamate and zinc ion.

The empirical formula and structural formula and molecular mass of mancozeb are given below:

Empirical Formula	*Structural Formula*	*Molecular Mass*
$[(CS_2NH\,CH_2)_2]_x - (Zn)_y$	 $\left(.\,S\overset{\overset{S}{\Vert}}{C}\;NHCH_2\,CH_2\;NH\overset{\overset{S}{\Vert}}{C}S.\;Mn.\right)_x (Zn)y$	271.02 with Manganese, 20 percent, *Min* and Zinc, 2.5 percent, *Min*

This standard was first published in 1978. This revision has been taken up in order to update the various requirements. In this revision the requirement of ethylene thiourea has been added.

In the formulation of this standard, due consideration has been given to the provisions of *Insecticides Act*, 1968 and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under the *Insecticides Act* and Rules, wherever applicable.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard
MANCOZEB, TECHNICAL — SPECIFICATION
(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for mancozeb, technical.

2 REFERENCES

The following standards contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1070 : 1992	Reagent grade water — Specification (*third revision*)
8069 : 1989	High density polyethylene (HDPE) woven sacks for packing pesticides— Specification (*second revision*)
8115 : 1976	Specification for double hessian jute bags for pesticides
8117 : 1976	Specification for DW tarpaulin laminated jute bags for pesticides
8190 (Part 1) : 1988	Requirements for packing of pesticides: Part 1 Solid pesticides (*second revision*)
10946 : 1996	Methods of sampling for technical grade pesticides (*first revision*)

3 REQUIREMENTS

3.1 Constituents

The material shall consist of mancozeb, technical with suitable stabilizer(s).

3.2 Physical

3.2.1 *Description*

The material shall be homogenous yellow or yellowish green powder, free from extraneous matter.

3.3 Chemical

The material shall also comply with the chemical requirements given in Table 1.

4 PACKING

The material shall be packed in clean and dry containers made of mild steel or tinplate or fibre board lined with polyethylene of thickness not less than 0.062 mm or double hessian jute bags (*see* IS 8115) or HDPE woven sacks (*see* IS 8069) or DW tarpaulin laminated jute bags (*see* IS 8117). The packing shall also meet the general requirements given in IS 8190 (Part 1).

5 MARKING

5.1 The containers shall be securely closed and the following information shall be marked legibly and indelibly on each container in addition to any other information as is necessary under the *Insecticides Act*, 1968 and Rules framed thereunder:

a) Name of the material;
b) Name and address of the manufacturer;
c) Batch number;
d) Date of manufacture;
e) Date of expiry;
f) Net quantity;
g) Nominal mancozeb content, percent (*m/m*);
h) A cautionary notice as worded in *Insecticides Act*, 1968 and Rules framed thereunder; and

Table 1 Requirements for Mancozeb, Technical

(*Clause* 3.3)

Sl No. (1)	Characteristic (2)	Requirement (3)	Method of Test, Ref to Annex (4)
i)	Mancozeb content, percent by mass, *Min*	85.0	A
ii)	Zinc content of mancozeb content, percent by mass, *Min*	2.5	B
iii)	Manganese content of mancozeb content, percent by mass, *Min*	20.0	C
iv)	Ethylene thiourea content, percent by mass, *Max*	0.5	D

j) Any other information required under the *Standards of Weights and Measures (Packaged Commodities) Rules*, 1977.

5.2 BIS Certification Marking

The product may also be marked with the Standard Mark.

5.2.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of the conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

Representative samples of the material shall be drawn according to IS 10946.

7 TESTS

7.1 Tests shall be carried out by the methods referred to in **3.2** and **3.3**.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and reagent grade water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Sl No.* (i)]

DETERMINATION OF MANCOZEB CONTENT

A-1 GENERAL

Mancozeb content may be determined by quantifying carbon disulphide content, generated during acidic digestion.

A-2 MANCOZEB DIGESTION AND EVOLUTION OF CARBON DISULPHIDE

A-2.1 Principle

Mancozeb is digested with sulphuric acid and the liberated carbon disulphide is allowed to react with alcoholic potassium hydroxide. Potassium methyl xanthogenate so produced is titrated with iodine.

A-2.2 Apparatus

A-2.2.1 *Balance (Analytical) and Carbon Disulphide Assembly*—The component parts are assembled as shown in Fig. 1, 2, 3 and 4. It consists of a 125 ml round bottom decomposition flask supported over a heating mantle by means of a clamp. The flask is fitted with a condenser. The side arm of distillation head is connected to the side arm of the lead acetate trap using rubber tubing. The top portion of lead acetate trap is fitted with side arm of potash scrubber through ball and socket joint. Little coating of grease may be applied to the surfaces of the joints and further it may be reinforced with clamp. The body of the potash absorber is connected to the vacuum system by means of rubber tubing. The aspiration rate is controlled by means of a pinch-cock (*see* Fig. 4).

NOTE — Do not use silicone rubber tubing for connecting the side arm of distillation head and lead lacetate trap. Silicone rubber has tendency to absorb carbon disulphide and lower results will be observed.

A-2.2.2 *Glass Vial* — Diameter 8-12 mm, height 20-25 mm for weighing mancozeb powder.

A-2.2.3 *Water Circulation System* — A suitable water circulation system is provided to circulate cooling water in the condenser as well as water jacket. The temperature of the cooling water is maintained so as to maintain the temperature of water jacket at 18-22°C and complete reflux in the condenser through out the experiment. Usually chilled water 8-10°C should be passed through condenser in parallel connected to common header of water inlet and outlet of this may be distributed through jacket in series.

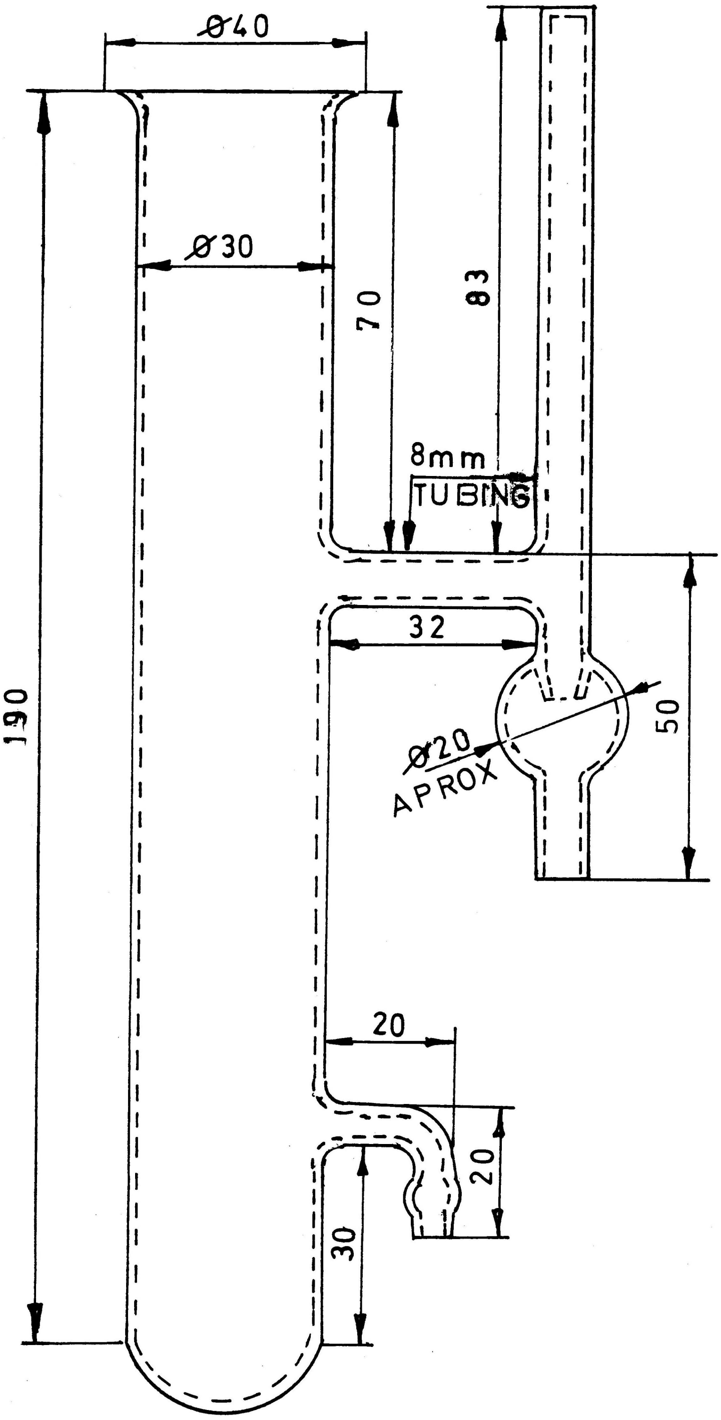
A-2.2.4 *Vacuum Pump*

A-2.3 Reagents

A-2.3.1 *Acetic Acid, 30 Percent Solution* — It is prepared by adding 3 volumes of AR grade acetic acid to 7 volumes of distilled water.

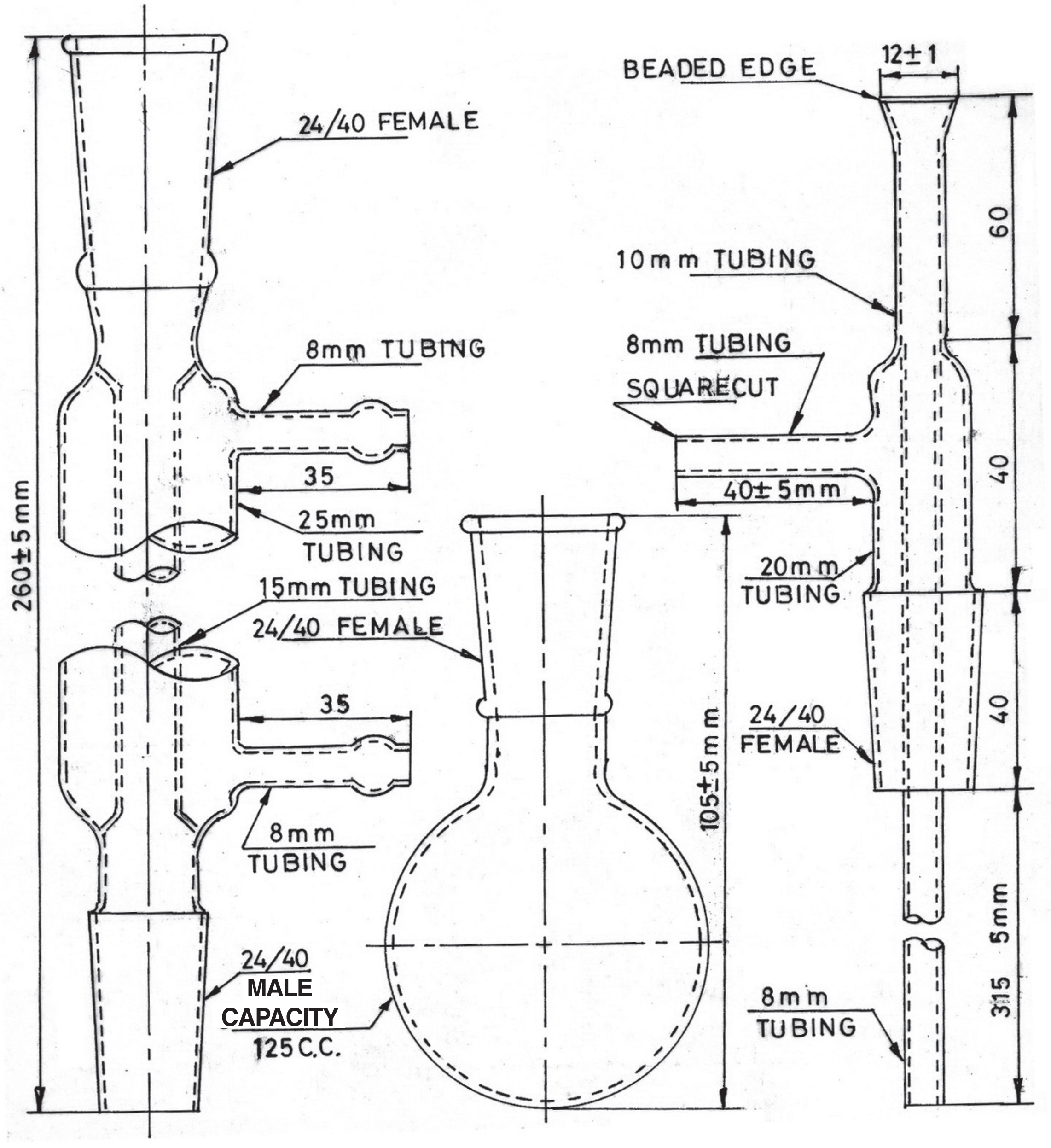
A-2.3.2 *Iodine, 0.1 N Solution* — It is prepared and standardized against analytical reagent grade $As_2 O_3$.

A-2.3.3 *Lead Acetate, 10 Percent Solution* — Dissolve 20.0 ± 0.1 g of reagent grade lead acetate trihydrate in 180 ml of distilled water.



All dimensions in millimetres.

FIG. 1 JACKET FOR POTASH SCRUBBER



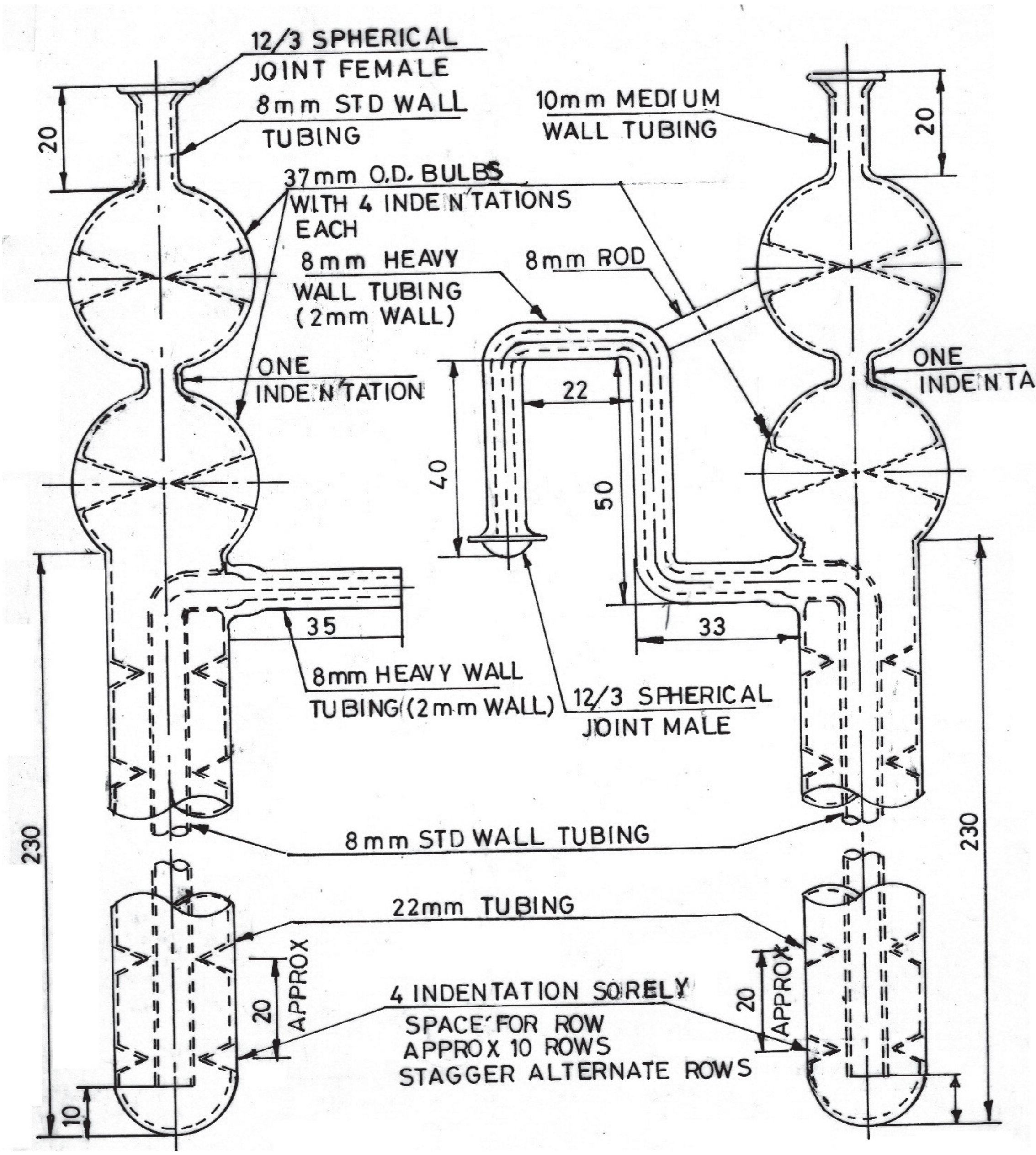
CONDENSER

DECOMPOSITION FLASK

DISTILLATION HEAD

All dimensions in millimetres.

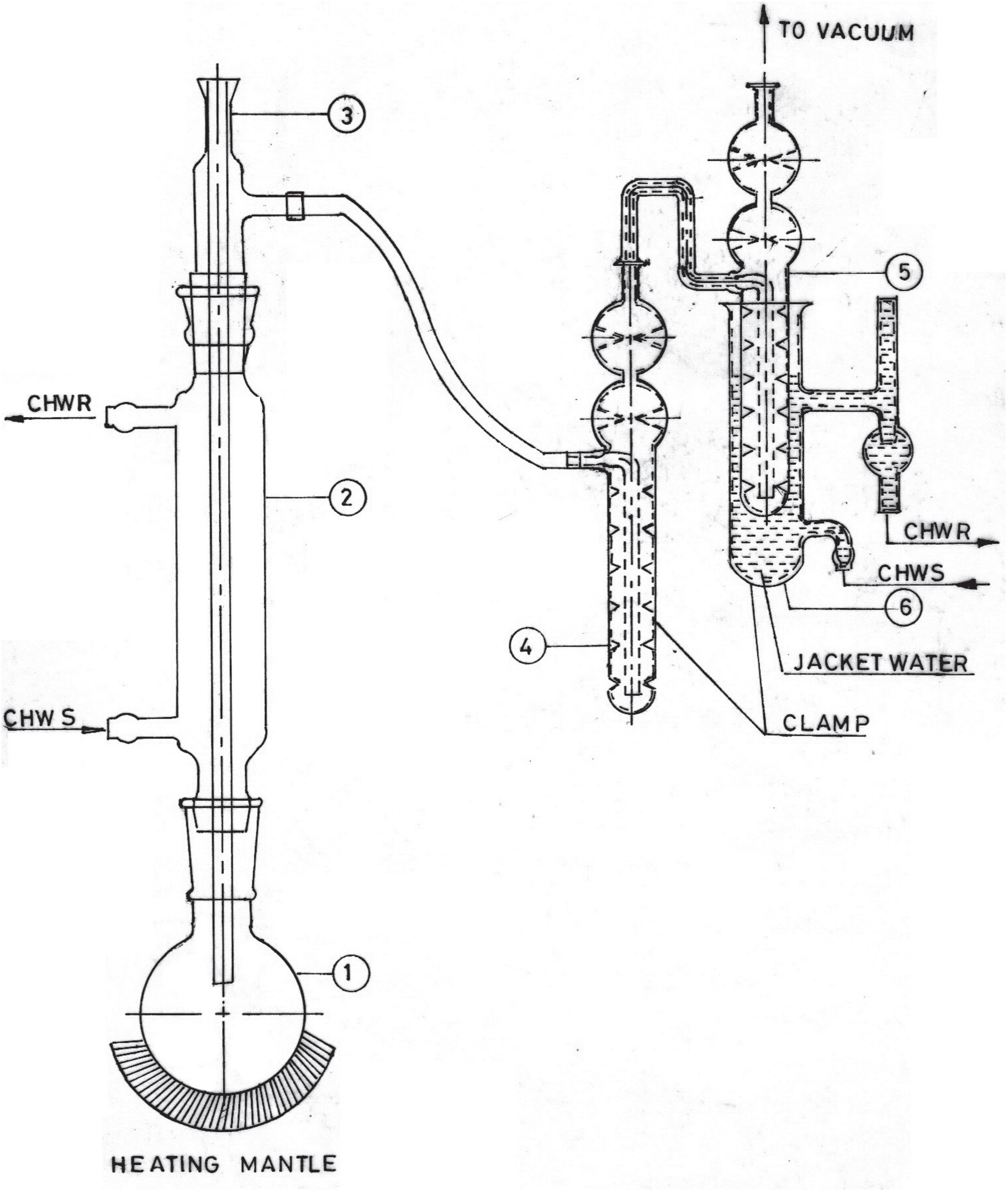
FIG. 2 CONDENSER, DECOMPOSITION FLASK AND DISTILLATION HEAD


12/3 SPHERICAL JOINT FEMALE
8mm STD WALL TUBING
20
37mm O.D. BULBS WITH 4 INDENTATIONS EACH
10mm MEDIUM WALL TUBING
20
8mm HEAVY WALL TUBING (2mm WALL)
8mm ROD
ONE INDENTATION
ONE INDENTATION
22
40
50
35
33
8mm HEAVY WALL TUBING (2mm WALL)
12/3 SPHERICAL JOINT MALE
230
230
8mm STD WALL TUBING
22mm TUBING
APPROX
20
APPROX
20
4 INDENTATION SORELY SPACE FOR ROW APPROX 10 ROWS STAGGER ALTERNATE ROWS
10

LEAD ACETATE TRAP **POTASH ABSORBER**

All dimensions in millimetres.

FIG. 3 LEAD ACETATE TRAP AND POTASH ABSORBER



All dimensions in millimetres.

FIG. 4 ASSEMBLY FOR THE DETERMINATION OF MANCOZEB CONTENT

A-2.3.4 *Phenolphthalein Indicator* — 0.5 percent ethanolic solution.

A-2.3.5 *Potassium Hydroxide, 2 N Methanolic Solution* — The solution should not be more than 2 days old. Dissolve 330 g of reagent grade potassium hydroxide pellets in 2.5 litre methanol, mix thoroughly and filter through filter paper.

A-2.3.6 *Starch Indicator*

A-2.3.7 *Sulphuric Acid, 1.1 N Solution* — Dissolve 75 ml of concentrated sulphuric acid (sp.gr. 1.84) in 2 350 ml distilled water and mix thoroughly.

A-2.4 Procedure

A-2.4.1 *Determination of Blank*

Pipette 25 ml of the 2 N methanolic potassium hydroxide solution into a 500 ml wide mouth Erlenmeyer flask which contains 250 ml chilled distilled water. Add 3 drops of phenolphthalein indicator solution and titrate with 30 percent acetic acid solution till just neutral. Add 5 ml starch indicator solution and titrate against 0.1 N iodine. If more than 0.1 ml of iodine solution is consumed, the methanolic potassium hydroxide solution cannot be used for the determination of mancozeb content.

A-2.4.2 If necessary, clean the apparatus thoroughly as follows.

A-2.4.3 Add several drop of concentrated hydrochloric acid to the lead acetate trap to dissolve any black lead sulphide that may be present, rinse the trap with tap water, then with acetone, then dry it with a jet of air. Clean the potash absorber, first by rinsing it with tap water then the distillation head and the decomposition flask with tap water and allow to drain.

A-2.4.4 Place sufficient 1.1 N sulphuric acid (about 55 ml for each determination) in a beaker and heat to 60°C on a hot plate. Do not allow the acid to concentrate (by evaporation) as this may cause erroneous results. The temperature of sulphuric acid should be 60 ± 5°C at the time of transferring to digestion flask.

A-2.4.5 Insert the distillation head into the condenser.

A-2.4.6 By pipette, transfer 25.0 ml of 10 percent lead acetate solution to the lead acetate trap (Absorber No. 1) through side arm.

A-2.4.7 By pipette, transfer 25.0 ml of 2 N methanolic potassium hydroxide solution to the potash absorber (Absorber No. 2) through top.

A-2.4.8 Connect the lead acetate trap to the distillation head. Insert the potash absorber into the coolant jacket then connect the top of the potash absorber to the vacuum system.

NOTE — Sample weight, length of digestion time, temperature of the potash absorber, etc, all contributing factors to erroneous results. Analysts must adhere to these particular procedure details.

A-2.4.9 Transfer 0.2 to 0.3 g of sample to an accurately weighed (to 0.000 1 g) clean any dry glass vial. Wipe the outside of the vial with a tissue and re-weigh the vial plus sample accurately to determine the exact weight of the sample. Place the vial in the 125 ml flask and connect it back to the assembly. Close the pinch-cock and connect the assembly to the vacuum line. Slowly, open the pinch-cock and adjust the rate of bubbling in the absorbers to 3 to 6 bubbles per second. Turn on the cooling water and maintain the temperature of the water jacket at 18-22°C through out the experiment. By graduated cylinder add carefully 50 ml of hot temperature (60 ± 5°C) 1.1 N sulphuric acid through the inlet tube of the distillation head. Make sure that the inlet tube dips in the liquid. Turn on the heating mantle and heat the contents of the flask to the boiling point as rapidly as possible, being careful not to allow the solution to go into the condenser (Sufficient chilling water in condenser enable 'Complete Reflux' of water vapour). Continue the heating and bubbling for exactly 1 h and 45 min. After the digestion is complete, quickly disconnect the potash trap and rinse the contents into 500 ml Erlenmeyer flask, with about 250 ml chilled distilled water. Add 3 drop of phenolphthalein indicator solution and titrate the solution with 30 percent acetic acid until the solution is just neutral. Titrate the neutralized solution with 0.1 N iodine solution till the colour changes by using starch indicator within 60 s otherwise discard the solution.

A-2.5 Calculation

$$\text{Active ingredient, percent by mass} = \frac{(V_1 - V_2) \times N \times F}{m}$$

where

V_1 = volume of iodine solution required for the sample, in ml;

V_2 = volume of iodine solution required for the blank, in ml;

N = normality of the iodine solution;

F = factor = 13.551 for mancozeb; and

m = mass of the sample taken for the test, in g.

ANNEX B

[*Table* 1, *Sl No.* (ii)]

DETERMINATION OF ZINC CONTENT IN MANCOZEB

B-1 GENERAL

Zinc content in mancozeb may be determined by complexation titration with EDTA and masking agent potassium cyanide.

B-2 ZINC CONTENT BY COMPLEXATION TITRATION

B-2.1 Principle

Since mancozeb contains manganese and zinc, both the metal ions, the latter is first masked with the help of potassium cyanide (KCN) and titrated against EDTA using Eriocrome Black-T.

After the appearance of first end point (V_1) the zinc ion is de-masked and the mixture is further titrated against EDTA to reach the end point (V).

The difference between V and V_1 is accounted for zinc content.

B-2.2 Apparatus

B-2.2.1 *Burette* — 50 ml/25 ml capacity having least count 0.05 ml.

B-2.2.2 *Conical Titration Flask* — 500 ml capacity.

B-2.3 Reagents

B-2.3.1 *Ascorbic Acid L* (+) — Reagent grade.

B-2.3.2 *Buffer Solution* — *p*H 10.

B-2.3.3 *EDTA Solution* — 0.1 M solution.

B-2.3.4 *Eriochrome Black T* — Calcon indicator powder (0.5 percent).

B-2.3.5 *Formaldehyde* — 37 percent solution.

B-2.3.6 *Potassium Cyanide Solution* — 5 percent (*m/m*).

B-2.4 Procedure

B-2.4.1 Weigh accurately sufficient quantity of the material to contain about 0.02 g zinc. Transfer it quantitatively, to a 500 ml conical flask. Add 50 ml of distilled water and 1.0-1.5 g of ascorbic acid. Swirl the flask to dissolve the contents. By graduated cylinder add 25 ml buffer solution and 10 ml potassium cyanide solution to the flask. Swirl the flask to mix the contents, then without delay titrate the solution with EDTA solution. View the titration by transmitted light from a day light type fluorescent light source. When approximately 20 ml of EDTA has been added, note if there are any granules present in the solution. If necessary, heat the solution to dissolve or disperse the granules and cool the solution. Then, add 10 ml buffer solution and a small quantity (approximately 10 mg) of Eriochrome Black T-Calcon Indicator to the solution. The solution will turn reddish purple in colour. Continue to titrate with EDTA solution to a bluish green end point that persists for at least 30 s. Record the ml required as V_1. Do not refill the burette. By graduated cylinder, add 5 ± 1 ml formaldehyde solution. The return of the original reddish purple colour indicates the presence of zinc. Continue the titration to a permanent bluish green end point. Record the total ml of EDTA solution used as V.

B-2.5 Calculation

B-2.5.1 Zinc, percent by mass = $\dfrac{(V - V_1) \times M \times 6.538}{m}$

where

V = end point (*see* **B-2.1**);
V_1 = first end point (*see* **B-2.1**);
M = molarity of EDTA solution; and
m = mass, in g, of the sample taken for the test.

B-2.5.2 Calculate zinc content in mancozeb by multiplying the value of mancozeb content to zinc percent.

ANNEX C

[*Table* 1, *Sl No.* (iii)]

DETERMINATION OF MANGANESE CONTENT IN MANCOZEB

C-1 GENERAL

Manganese content in mancozeb may be determined by complexation titration with EDTA and masking agent potassium cyanide.

C-2 MANGANESE CONTENT BY COMPLEXATION TITRATION

C-2.1 Principle — See **B-2.1**.

C-2.2 Apparatus — See **B-2.2**.

C-2.3 Reagents — See **B-2.3**.

C-2.4 Procedure

C-2.4.1 Weigh accurately about 1.00 g of sample into a 500 ml wide-mouth conical flask. By graduated cylinder, add 50 ml of de-ionized water and swirl to completely suspend the sample. Add approximately 1.0 to 1.5 g of ascorbic acid by graduated cylinder; add 25 ml of *p*H 10 buffer solution and 10 ml of 5 percent potassium cyanide solution. Swirl to mix, then without delay titrate the sample with the standard 0.1 M solution of EDTA. When approximately 20 ml of EDTA have been added, note if there are any granules present in the solution. If necessary, heat the solution to dissolve or disperse the granules, cool, then add 10 ml (graduated cylinder) buffer solution. Add a small quantity (approximately 10 mg) of Eriochrome Black T-Calcon indicator powder on the end of a spatula; the sample suspension will turn reddish purple in colour. Continue to titrate with EDTA solution to the end point. At the end point all of the sample will be dissolved and the solution will have a blue-green colour that will persist for at least 30 s. Record the ml required as titer *A*.

C-2.5 Calculation

$$\text{Manganese content, percent by mass} = \frac{\text{Titer } A \times M \times 5.493}{m}$$

where

M = molarity of EDTA solution; and

m = mass of the sample taken for the test, in g.

C-2.5.1 Calculate manganese content in mancozeb by multiplying the value of mancozeb content to manganese percent.

ANNEX D

[*Table* 1, *Sl No.* (iv)]

DETERMINATION OF ETU IN MANCOZEB, TECHNICAL

D-1 PRINCIPLE

The ethylene thiourea (ETU) content is determined by high performance liquid chromatography (HPLC) using the external area method.

D-2 APPARATUS

D-2.1 High Performance Liquid Chromatograph — A high performance liquid chromatograph (HPLC) equipped with UV detector and coupled to a chromatographic software/printer plotter-*cum*-integrator. The suggested operative parameters are as follows, but can be changed, if necessary, provided standardization is done:

Column	:	Stainless Steel, 250 mm × 4.6 mm internal diameter, packed with C-18 of 5 µm particle size
Mobile phase	:	Triple distilled water containing 0.05 percent *v/v* tetrahydrofuran (Filter the solution through a membrane filter that is nylon 66 of 0.45 µm)
Flow rate	:	1.0 ml/min
Detector	:	UV at 233 nm
Injection volume	:	20 µl
Temperature	:	Ambient
Retention time (Guidance value)	:	5.4 min (approximately)

D-2.2 Analytical Balance — L.C. 0.1 mg.

D-2.3 Ultrasonic Bath

D-2.4 Syringe — 20 µl capacity.

D-2.5 Sintered Glass Crucible — G-4 sintered glass crucible.

D-2.6 Glassware — Standard volumetric flasks of

25 ml, 50 ml, 100 ml and pipettes of 1 ml, 2 ml.

D-3 REAGENTS

D-3.1 Ethylene Thiourea — Reference standard of known purity.

D-3.2 Tetrahydrofuran — Analytical reagent grade or equivalent.

D-3.3 Triple Distilled Water

D-4 PROCEDURE

D-4.1 Preparation of Standard Solution

Weigh accurately 70-75 mg of ETU standard (M_1) of known purity in a 100-ml volumetric flask. Dissolve the contents and make up the volume up to the mark with triple distilled water (Stock solution *A*). Pipette out 1 ml of stock solution A to another 100-ml. volumetric flask and make up the volume with triple distilled water (Stock solution *B*).

D-4.2 Preparation of Sample Solution

Weigh accurately about 3.0 g of sample (M_2) in a 100 ml volumetric flask. Add about 40 ml triple distilled water and sonicate for 10 min. Make up the volume using triple distilled water. Filter the solution through sintered glass crucible to get a clear filtrate. Pipette out 2 ml of above solution in another 50 ml volumetric flask and dilute upto the mark with triple distilled water (Solution *S*).

NOTE — Solutions A, B, and S should be discarded after the testing is over. These solutions cannot be used next days. It gives inaccurate results.

D-5 ESTIMATION

Inject the standard solution consecutively, until the area of reference substance of two successive injections do not deviate from mean by more than 2 percent. Then inject the sample solution. The sequence of injections then to be followed is as follows:

$$\ldots C\, S_1\, S_1\, C\, S_2\, S_2\, C\, S_3\, S_3 \ldots C \ldots\ldots$$

where

C = standard solution, and

S = sample solution (1, 2, 3,…………..*n*).

From the chromatograms of the standard solution and sample solution, measure the peak areas of ethylene thiourea and calculate the percentage of ethylene thiourea as given in **D-6**.

D-6 CALCULATION

$$\text{Ethylene thiourea content, percent, } m/m = \frac{M_1 \times A_2 \times P}{M_2 \times A_1 \times 4}$$

where

M_1 = initial mass of standard ethylene thiourea, in g;

M_2 = initial mass of sample, in g;

A_1 = area of ethylene thiourea peak in the chromatogram of standard solution;

A_2 = area of ethylene thiourea peak in the chromatogram of sample solution; and

P = percent purity of standard ethylene thiourea.

NOTE — Adjust the sample size, if area of sample and standard do not match within 50 percent range of each other.

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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards : Monthly Additions'.

This Indian Standard has been developed from Doc No.: FAD 1 (1352).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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